

<p>98-090084/09 A60 E11 (A26) <b>TAKE 96.01.29</b>  TAKEDA CHEM IND LTD *JP 09208589-A  96.01.29 96JP-012933 (97.08.12) C07F 7/10, 7/18 // C08G 77/388  Preparation of isocyanate compounds with alkoxy:silyl groups -  comprises reacting alkoxy:silyl:alkyl primary amine with  phosgene in inert organic solvent in presence of alkali or alkaline  earth metal compound  <b>C98-030549</b></p>	<p>A(10-E22A) E(5-E2, 5-E3)</p>
<p>Preparation of isocyanate compounds of formula (II) comprises  reacting an alkoxy:silyl-alkyl primary amine of formula (I) with  phosgene in an inert organic solvent in the presence of an alkali metal  or alkaline earth metal compound.</p> $R_2-Si(R_1)(R_3)-(OSi(R_4)(R_5))_n-R_6-NCO \quad (II)$ $R_2-Si(R_1)(R_3)-(OSi(R_4)(R_5))_n-R_6-NH_2 \quad (I)$ <p><math>R_1</math>-<math>R_3</math> = hydrocarbon group or alkoxy;  <math>R_4</math>, <math>R_5</math> = hydrocarbon group, alkoxy or alkoxy-siloxy;  <math>R_6</math> = 1-8C alkylene; and  <math>n</math> = integer of 0-8.  At least one of <math>R_1</math>-<math>R_5</math> is alkoxy.</p>	<p><u>USE</u> Used as silicon modifiers for organic compounds.</p> <p><u>ADVANTAGE</u> Hydrochloric acid generated during the reaction with phosgene is efficiently removed by low cost raw materials, and side reactions avoided even in a distillation step. The low temperature reaction step necessary in the prior art is avoided by using tertiary amines. (II) are produced in high yield.</p> <p><u>PREFERRED PREPARATION</u> The alkali metal or alkaline earth metal compound is weak acid salt or oxide (specifically, CaO). The inert organic solvent is a polar solvent.</p> <p><u>EXAMPLE</u> A 500 ml flask with a reflux condenser, dropping funnel and stirrer contained a solution of 9.9 g phosgene in 300 g EtOAc, to which was added 16.8 g CaO. A solution of 22.1 g <math>\gamma</math>-  JP 09208589-A+</p>

triethoxysilylpropylamine in 70 g EtOAc at 0-10°C was dropwise added over 1 hour. The mixture was warmed to 50°C and matured for 1 hour. The resulting CaCl<sub>2</sub> and excess CaO were filtered off, and the filtrate after removal of EtOAc was distilled to give 23.5 g colourless clear liquid having b.pt. of 92-94°C/5.0 mmHg. Gas chromatography indicated the product was  $\gamma$ -triethoxysilyl propylisocyanate (yield: 95.1%; purity: 99.5%). (HW)  
(5pp052DwgNo.0/0)

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